

Surfactant induced smooth and symmetric interfaces in Cu/Co multilayers

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In this work we studied Ag surfactant induced growth of Cu/Co multilayers. The Cu/Co multilayers were deposited using Ag surfactant by ion beam sputtering technique. It was found that Ag surfactant balances the asymmetry between the surface free energy of Cu and Co. As a result, the Co-on-Cu and Cu-on-Co interfaces become sharp and symmetric and thereby improve the thermal stability of the multilayer. On the basis of obtained results, a mechanism leading to symmetric and stable interfaces in Cu/Co multilayers is discussed.

I. INTRODUCTION

Smooth and symmetric interfaces of magnetic multilayers are of much interest because of their application in technological devices like recording media, read heads and sensors.¹⁻³ The magnetic layers separated by the non magnetic spacer layer show the giant magnetoresistance (GMR) due to oscillatory exchange coupling for varying spacer layer thicknesses.^{4,5} Thickness fluctuation of the spacer layer due to the interface roughness affect the strength of exchange coupling.⁶ GMR largely depends on the asymmetry of the roughness at the interface due to the spin dependent scattering across the interfaces.⁷⁻⁹ It was experimentally evidenced that sputtered Cu/Co multilayer exhibit the largest GMR with oscillatory exchange coupling.⁵ The GMR effect was first observed in Fe/Cr superlattices¹⁰ and experimental and theoretical studies demonstrate that GMR in Fe/Cr multilayer¹¹ increases with the presence of roughness at magnetic/nonmagnetic interface while it decreases with interface roughness for Cu/Co multilayer.¹² Therefore smooth and symmetric interfaces in Cu/Co multilayer are essential for application point of view.

Asymmetric interface occurs in multilayers due to the difference in the surface free energy (γ) of the elements.¹³ In Cu/Co multilayer system γ for Cu and Co is different. As such the surface free energy depends on the crystallographic orientation, in case of polycrystalline structures, γ for average face is relevant. The experimentally observed values of γ for the average face of Cu, Co and Ag are 1.8 Jm^{-2} , 2.55 Jm^{-2} and 1.24 Jm^{-2} , respectively.^{14,15} This difference in the surface free energy leads to wetting of Cu-on-Co and de-wetting of Co-on-Cu, giving rise to a smooth Cu-on-Co and a rough Co-on-Cu interface. This was experimentally evidenced by Timothy *et al.* in scanning tunnelling microscopy demonstrating that Co make islands over Cu while Cu make a smooth layer on Co in Cu/Co multilayers.¹⁶

It has been demonstrated in the literature that by using a surface active species or so called *surfactant* the difference between the surface free energies can be minimized. Use of such surfactants in the crystal growth technology is well established.¹⁷ However, in case of thin films deposited in vacuum, surfactants have been used only in few multilayer systems.¹⁸⁻²⁵ Egellhoff and Steigerwald¹³ studied the role of adsorbed gases (H, O, N, Co and S) in deposition of metal-on-metal epitaxial systems. These adsorbed gases float or segregate to the surface, balancing the surface/interface energy and strain during the growth. Gaseous surfactants such as oxygen, also suppresses the intermixing and increases the GMR by restricting pinholes in Cu spacer layer during deposition.²⁶⁻²⁸ Although, due to enhanced mobility of gaseous surfactants, it is likely that they get trapped across the grain boundaries.

In case of Ge/Si(100) multilayers, Copel *et al.* have demonstrated that use of As surfactant triggers the layer-by-layer type growth and inhibits interdiffusion.¹⁸ Hoegen *et al.* have shown that Sb surfactant not only inhibits interdiffusion but results in a relaxed (strain free) and defect free Ge film on Si(111).²⁹ In a theoretical study by Barabási the interaction of surface with a surfactant was described.³⁰ In an another theoretical work by Zhang and Lagally³¹ the surfactant mediated layer-by-layer growth was described on the basis of atomic interactions. Recently, Egellhoff and co-workers have explored the effect of surfactant (e.g In, Ag, O, Pb etc.) in Cu based spin valve systems and demonstrated that surfactant improves the surface and interface property as well as increases the GMR value.^{19,32,33} In an another study, Camarero *et al.* have demonstrated that Pb atoms used as surfactant suppresses the twin formation which increases the coupling between Co layers.²³ Theoretical studies also show that monolayer of Pb used as a surfactant in Cu/Co multilayer minimizes the difference in surface free energy of Cu and Co, inhibits the island formation and floats over the surface by atomic exchange process.^{34,35} The use of

Ag surfactant was also studied to examine the interfacial intermixing^{36,37} and GMR in magnetic multilayers.³⁸ In case of Ti/Ni multilayers Ag surfactant was used to get smooth and symmetric interface.³⁹ However, detailed studies on the Ag surfactant mediated growth in Cu/Co multilayers have not been performed.

In the present work we studied the effect of Ag surfactant in Cu/Co multilayer. It was also investigated how this addition of Ag surfactant affects the structural and magnetic properties of Cu/Co multilayers. We used neutron reflectivity (NR) technique which is non destructive and measures the interface roughness with an accuracy less than an Å. In addition, we used NR to measure the interdiffusion across Cu/Co interfaces. It was found that addition of Ag surfactant makes Cu-on-Co and Co-on-Cu interfaces smooth and symmetric which are otherwise rough and asymmetric. This leads to reduced interdiffusion and thereby improved thermal stability of Cu/Co multilayers prepared using Ag surfactant. The obtained results are presented and discussed in the following sections.

II. EXPERIMENTAL DETAILS

Ion beam sputtering (IBS) technique was used to deposit the Cu/Co multilayers with and without Ag surfactant. The Ar⁺ ions of energy 1 keV were produced using a radio-frequency ion beam source (Veeco 3cm RF source). The ion beam was neutralized using a RF generated electron flood source. The ion beam of size 3 cm was kept incident at an angle of 45° with respect to a target. The targets were mounted on a rotary motion feedthrough which can hold up to four different targets. The targets were sputtered alternatively to deposit a multilayer structure. The samples were prepared without any surfactant as a reference (sample A) and with Ag surfactant (sample B) added on top of a Cu buffer layer deposited on a Si(100) substrate at room temperature (without intentional heating). The nominal structure of samples are given below:

- (A) Cu (10 nm)/[Cu (3 nm)/Co (2 nm)]₁₀
- (B) Cu (10 nm)/Ag (0.2 nm)/[Cu (3 nm)/Co (2 nm)]₁₀

Here, the Cu layer thickness of 3 nm corresponds to the third (and weakest) AF peak in the oscillatory exchange coupling. At this thickness it is expected that magnetoresistance (MR) will be small compared to the first or second maxima around 1 nm and 2 nm, respectively.⁵ The Cu layer thickness of 3 nm was chosen as at lower thickness the Bragg peak in neutron reflectivity (NR) will appear at high q_z values and due to limited flux of neutron sources it may be difficult to do NR measurements in a reasonable time.

Prior to the deposition of samples the base pressure was about $2 \cdot 10^{-8}$ mbar and during deposition the pressure was about $5 \cdot 10^{-4}$ mbar due to flow of Ar gas (purity 99.9995%) in the source and neutralizer. X-ray and neutron reflectivity measurements were carried out to

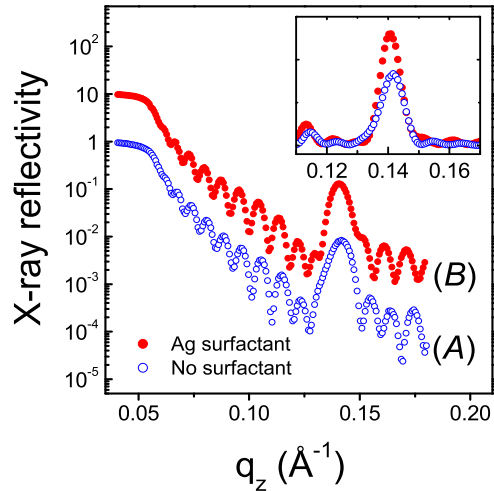


FIG. 1. (color online) X-ray reflectivity of Cu/Co multilayers deposited without surfactant (A) with Ag surfactant (B). Inset show the Bragg peak intensity at Bragg peak. The pattern on y-axis have been shifted for clarity.

measure the thickness, surface and interface roughness of the samples. X-ray reflectivity (XRR) measurements were carried out using x-rays of wavelength 1.54 Å generated using a laboratory source. The NR measurements were carried out at the SuperADAM instrument at ILL, Grenoble, France using neutrons of wavelength 4.4 Å. In order to study the thermal stability of the samples, the NR measurements were also carried out at NARZISS reflectometer at SINQ/PSI. X-ray diffraction (XRD) measurements were carried out with 1.54 Å x-rays in the θ -2 θ geometry using a standard diffractometer (Bruker D8 Advance) equipped with a fast 1-D detector based on silicon drift technology (Bruker LynxEye).

The magnetoresistance (MR) measurement for all the samples were carried out at room temperatures using four point probe method. The direction of the current flowing in the sample was along the direction of the magnetic field (parallel to the surface of the sample). Here, the MR is defined as $MR = (R_o - R_{sat})/R_o$, where R_o is the resistance in the absence of magnetic field and R_{sat} is the resistance under the magnetic field in which the sample are magnetically saturated.

III. RESULTS

A. X-ray and neutron reflectivity measurement

Fig. 1 shows the x-ray reflectivity pattern of the as-deposited samples. The XRR pattern for the samples A is shown in (fig. 1 A) and for the sample B in (fig. 1 B). As can be seen from the figure, the XRR pattern shows a Bragg peak around $q_z = 0.14 \text{ Å}^{-1}$, corresponding to the

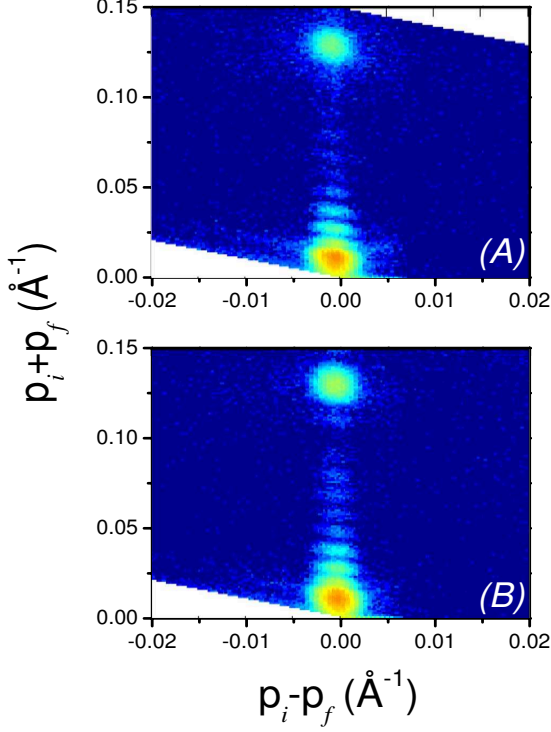


FIG. 2. (color online) 2-D Position sensitive detector (PSD) image of Cu/Co multilayers prepared without surfactant (A) and with Ag surfactant (B).

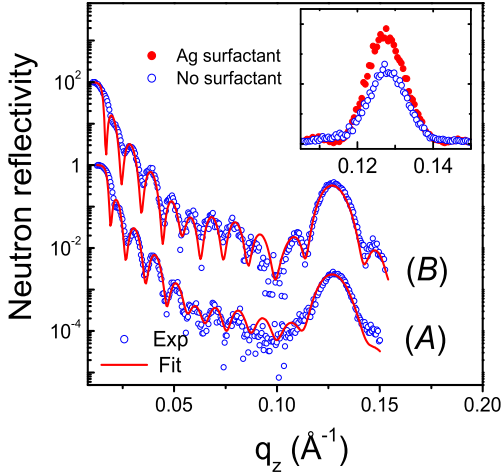


FIG. 3. (color online) Neutron reflectivity pattern Cu/Co multilayers prepared without surfactant (A) and with Ag surfactant (B). Inset shows the reflectivity at Bragg peak. The pattern on y-axis have been shifted for clarity.

bilayer period of 4.9 nm which is close to the nominal value of 5 nm. The oscillation in the pattern correspond to the total thickness of the sample. As can be seen from the inset of fig. 1, the intensity at the position of Bragg peak is enhanced significantly when Ag surfactant was added to the multilayer structure. Such an

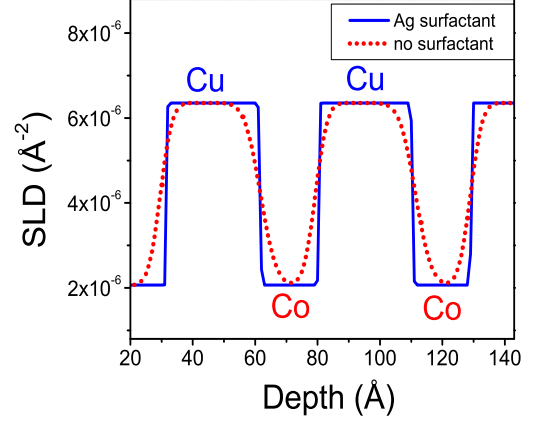


FIG. 4. (color online) Scattering length density profile of with surfactant (solid line) and without Ag surfactant (dash line) samples of Cu/Co multilayer.

enhancement in the intensity of the Bragg peak indicates that the interface get smoother with addition of Ag surfactant. It may be noted that the contrast between Cu and Co for the x-rays of wavelength used in this work is rather poor due to a small difference between the electron density of Cu and Co. For x-rays the refractive index is defined as $n = 1 - \delta - i\beta$ and with 1.54 Å x-rays the dispersion (δ) and absorption (β) parts of optical constants in terms of number density are: $\delta_{\text{Cu}} = 6.45 \cdot 10^{-5} \text{ Å}^{-2}$, $\beta_{\text{Cu}} = 1.45 \cdot 10^{-6} \text{ Å}^{-2}$, $\delta_{\text{Co}} = 6.30 \cdot 10^{-5} \text{ Å}^{-2}$, $\beta_{\text{Co}} = 9.135 \cdot 10^{-6} \text{ Å}^{-2}$. Therefore the Bragg peak appearing in fig. 1 is basically due to contrast in the absorption part of the refractive index which makes it rather difficult to model the x-ray reflectivity data. However, qualitatively it can be seen that addition of Ag surfactant helps in reducing the interface roughness. Therefore, in order to get more insight into the interfaces of the multilayers, we performed neutron reflectivity measurements. In case of neutrons the contrast between Cu and Co is much larger as compared to x-rays, with absorption being negligible due to much weaker interaction of neutrons with matter as compared to x-rays. The values of scattering length density (SLD) for Cu and Co in case of neutrons are $6.55 \cdot 10^{-6} \text{ Å}^{-2}$ and $2.26 \cdot 10^{-6} \text{ Å}^{-2}$, respectively.

The neutron reflectivity pattern of samples were recorded using a 2-D position sensitive detector (PSD). The PSD images of samples are shown in fig. 2 as a function of $(p_i + p_f)$ and $(p_i - p_f)$ with $p_{i(f)} = 2\pi \sin \theta_{i(f)} / \lambda$ the normal to surface component of the incoming (outgoing) wave vector and $\theta_{i(f)}$ the angle of the incidence (scattering) and λ being the wavelength of the neutrons.⁴⁰ A cut across the line for $p_i = p_f$ gives the specular reflectivity which is plotted in fig. 3 for samples A and B as a function of q_z ($q_z = p_i + p_f$). Here it may be noted that

in the NR pattern we did not observe magnetic Bragg peak which is expected due to antiferromagnetic (AF) coupling.⁴¹ This may be due to the fact that the thickness of the Cu layer correspond to the third AF peak which is weakest in the oscillatory exchange coupling. Further since we have only 10 repeats of Cu/Co bilayers, it is expected that the intensity of the AF peak will be too small to measure experimentally in this case. This is further confirmed by the MR measurements (shown later) where the typical values of MR are about 1% in the as-deposited samples.

As can be seen from the PSD images, the Bragg peak appears as a bright spot for both samples. The brightness of the Bragg reflected region is more intense for sample *B* as compared to sample *A*. This shows that with addition of Ag surfactant the reflectivity at the Bragg peak enhances as also observed with x-ray reflectivity data. Such an enhancement in the Bragg peak may happen due to a reduction in the interface roughness. If the roughness decreases, it should result in less diffuse scattering which appears along the x-direction ($p_i - p_f$) in fig. 2. A closer look at the PSD images indeed shows lesser diffuse scattering for sample *B* around the critical edge and Bragg peak positions. However, as shown later, the roughnesses are rather small therefore, the diffuse scattering expected from these multilayers is small.

Fig. 3 shows the deduced NR pattern for samples *A* and *B*. The patterns were fitted using Parratt's formalism⁴² and the fitted results are given in table I. As can be seen from the table, the value of reflectivity at the Bragg peak increases from 0.23% to 0.36% when Ag surfactant was added. The roughness of Co-on-Cu interface and Cu-on-Co interface was 0.38 nm and 0.18 nm, respectively when no surfactant was used. With addition of Ag surfactant the interface roughness of both interface becomes equal at about 0.1 nm. This value of roughness appears rather small, which may be due to the fact that we did not subtract the diffuse scattering. Therefore the value of roughnesses should be taken as a lower limit. Fig. 4 shows the SLD profile of samples *A* and *B*, obtained from the fitting of NR data. As can be seen from the figure, the SLD profiles were asymmetric and broad when no surfactant was used, with addition of Ag surfactant the profiles become sharp and symmetric. This clearly shows that the addition of Ag surfactant in the Cu/Co multilayer results in an appreciable decrease of interface roughnesses and also the of Co-on-Cu and Cu-on-Co interfaces become symmetric.

B. Thermal stability of Cu/Co multilayer

The thermal stability of the Cu/Co multilayer was studied by doing neutron reflectivity, x-ray diffraction and magnetoresistance measurements in the samples *A* and *B* after annealing the samples in a vacuum furnace with a base pressure of about $1 \cdot 10^{-6}$ mbar. The annealing of the samples was performed between 373 K to 673 K

TABLE I. Intensity at Bragg peak in Neutron reflectivity measurement of with and without Ag samples deposited by ion beam sputtering.

Sample	No surfactant	Ag surfactant
R_{Bragg} (%)	0.23 ± 0.01	0.36 ± 0.01
d (nm)	4.9 ± 0.1	4.9 ± 0.1
$\sigma_{[\text{Co-on-Cu}]}$ (nm)	0.36 ± 0.01	0.11 ± 0.01
$\sigma_{[\text{Cu-on-Cu}]}$ (nm)	0.18 ± 0.01	0.10 ± 0.01

with a step of 100 K. It was found that up to a temperature of 473 K the properties of samples remain identical to the as-deposited samples, however above this temperature, the samples prepared using Ag surfactant were found more stable as compared to samples prepared without any surfactant. The results presented in the following subsections:

1. Neutron reflectivity measurements

In order to check the thermal stability, the NR measurements were performed at the NARZISS reflectometer at SINQ/PSI. The neutron reflectivity pattern of the samples prepared with and without Ag as a surfactant are shown in fig 5. The measurements shown are in the as-deposited samples and after annealing the samples at 573 K, and at 673 K for 1 hour at each temperature. The Bragg peak corresponding to the multilayer period appears at nearly the same angles in both samples. The intensity of the Bragg peak is expected to decay due to the inter-diffusion as the annealing temperature is raised. This decay is seen to be faster in the samples when no surfactant was used.

In the sample *B*, the Bragg peak intensity decreases marginally at temperature of 573 K. However, in case of sample *A* where no Ag surfactant was used at 573 K there is significant decrease of Bragg peak intensity. At 673 K the Bragg peak completely disappears for the sample *A* while for the sample *B* the Bragg peak can still be seen, however its intensity is also significantly reduced. This result clearly demonstrates that the thermal stability of the Cu/Co multilayer is improved with Ag surfactant. From the measured neutron reflectivity data the inter-diffusion in both samples can be quantified and the decay of the Bragg peak intensity can be used to calculate the diffusion coefficient using the expression.^{43,44}

$$I(t) = I(0) \exp\left(-\frac{8\pi^2 D}{\ell^2} t\right), \quad (1)$$

where $I(0)$ is the intensity before annealing and $I(t)$ is the intensity after annealing time t at temperature T , ℓ is the bilayer periodicity. With known diffusion coefficient (D) calculated using eq. 1 the inter diffusion length (L_d) can be calculated with the expression $L_d^2 = 6Dt$ in the

2. X-ray diffraction measurements

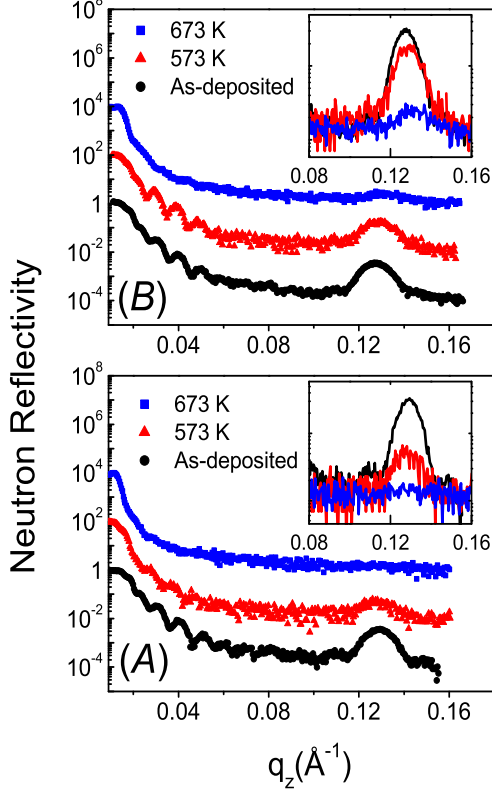


FIG. 5. (color online) Neutron reflectivity of Cu/Co multilayers prepared without any surfactant (A) and with Ag surfactant (B) after annealing at different annealing temperatures. The pattern were shifted on y-scale for clarity. Inset shows a comparison of the intensity at the Bragg peak.

TABLE II. Inter diffusion length (L_d) and diffusivity (D) obtained from Neutron reflectivity measurement of with and without Ag samples.

Sample	No surfactant		Ag surfactant	
	$D(m^2s^{-1})$	$L_d (nm)$	$D (m^2s^{-1})$	$L_d (nm)$
573 K	$1.9(\pm 0.3) \cdot 10^{-22}$	$2(\pm 0.2)$	$5.1(\pm 0.5) \cdot 10^{-23}$	$1.0(\pm 0.2)$
673 K	—	—	$2.8(\pm 0.3) \cdot 10^{-22}$	$2.5(\pm 0.3)$

direction normal to the surface of samples.⁴⁵ The inter-diffusion lengths obtained in this way are given in table II along with the values of diffusion coefficient D . Clearly, the inter-diffusion length L_d is significantly smaller for the sample prepared with Ag surfactant as compared to the sample prepared without any surfactant. Therefore, by using Ag surfactant the inter-diffusion and diffusivity in Cu/Co multilayers is reduced significantly.

The XRD pattern of Cu/Co multilayers prepared with and without Ag surfactant in the as-deposited state and after annealing at temperatures of 573 K and 673 K are shown in fig. 6. The XRD pattern of the as-deposited samples show a sharp peak around $2\theta = 43.6^\circ$ and a broader peak around $2\theta = 50.5^\circ$ corresponding to Cu (111) and Cu (200) reflections, respectively. After annealing at 573 K, these peaks shift towards the higher angle side both in sample A and B, which indicates a reduction in the inter-atomic distance. Further annealing at 673 K results in complete suppression of Cu (111) peak for the sample prepared without any surfactant, whereas in the sample prepared using Ag surfactant this peak remains intact. It is interesting to see that in the sample prepared without any surfactant a new peak appears at $2\theta = 45.6^\circ$ (shown as star in fig. 6), which does not correspond to any known phases of Cu or Co. As Cu-Co is an immiscible system, therefore this peak can not be assigned to a known alloy of CuCo. In order to confirm our results, we repeated this experiment by preparing a new Cu/Co multilayer sample both with and without surfactant and observed the same results as shown in fig. 6. It has been reported in the literature that an intermixed fcc-structured superlattice phase of CuCo may form upon solid-state interfacial reaction after annealing at moderate temperature around 573 K.⁴⁶ However, more details about such superlattice structure are not available.

From the interdiffusion measurements using neutron reflectivity as we observed that the interdiffusion in the sample prepared without any surfactant was significantly enhanced as compared to the sample prepared using Ag surfactant. An enhanced interdiffusion may give rise to an intermixed CuCo phase as observed in the present case. However, at 673 K the interdiffusion even in the sample prepared with surfactant is similar to the sample prepared without surfactant at 573 K, still no such intermixed CuCo phase can be observed in the sample prepared using Ag surfactant. As pointed out by Li *et al.*,⁴⁶ the origin of intermixed CuCo phase may be due to the excess surface free energy. In the case when a sample is prepared using Ag surfactant the surface free energy of Cu and Co are balanced and in this situation the excess free energy for formation of an intermixed CuCo phase may not be available. Therefore our results clearly demonstrate that by balancing the surface free energy using Ag surfactant the thermal stability of Cu/Co multilayers can be enhanced.

3. Magnetoresistance measurements

Fig. 7 shows the MR of Cu/Co multilayers prepared without surfactant (fig. 7 A) and with Ag surfactant (fig. 7 B). In case of sample A, the value of MR increases after annealing at 573 K and suppresses completely after annealing at 673 K. Whereas in case of sample B, the

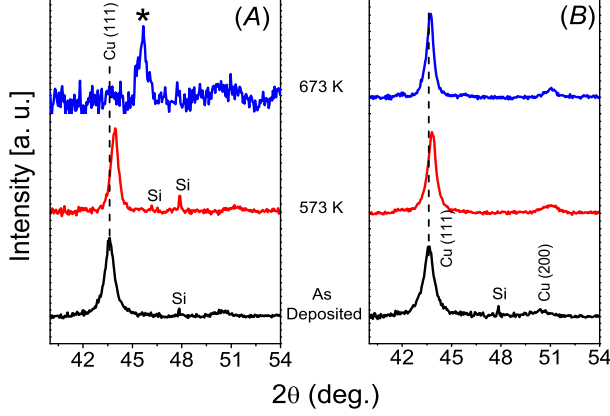


FIG. 6. (color online) XRD pattern of Cu/Co multilayers prepared without surfactant (A) and using Ag surfactant (B) at different annealing temperatures.

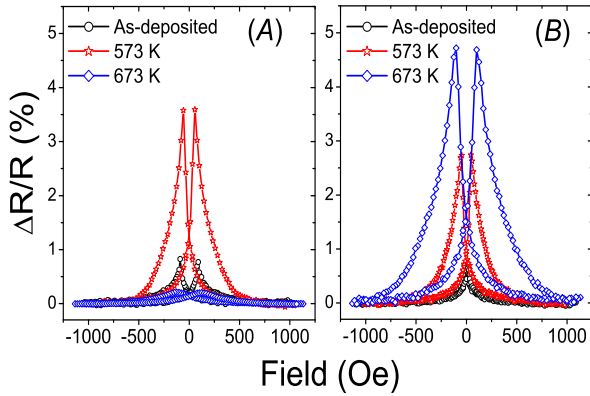


FIG. 7. (color online) MR of sample A and sample B at different annealing temperature.

value of MR increases both at 573 K and 673 K. The obtained MR results can be understood in correlation with NR and XRD results. As can be seen from the NR and XRD measurements, for the sample A, the Cu and Co layer interdiffuse appreciably at 573 K. Therefore an increase in the MR at 573 K may be due to formation of a granular structure as a result of interdiffusion of Cu and Co. In this situation the distance between Co atoms separated Cu atoms may get decreased which may result in an exchange coupling between Co atoms. However, at 673 K when an intermixed CuCo phase is formed, the MR suppresses completely due to formation of this intermixed phase. In the sample prepared using Ag surfactant since no such intermixed phase is formed, MR still increases at 673 K.

IV. DISCUSSIONS

From the results presented in the previous sections, the effect of Ag surfactant in the Cu/Co multilayer may be summarized as: (i) reduction of interface roughness leading to symmetry of Co-on-Cu and Cu-on-Co interfaces (ii) suppression of interdiffusion and (iii) improvement in the thermal stability of the multilayer.

As mentioned before $\gamma_{\text{Cu}}=1.8 \text{ J/m}^2$ and $\gamma_{\text{Co}}=2.55 \text{ J/m}^2$. This difference in the surface free energy leads to asymmetric Co-on-Cu and Cu-on-Co interfaces. When Cu gets deposited on Co, it will wet the surface of Co as the surface free energy of Cu is smaller. On the other hand when Co is getting deposited on Cu, it will de-wet or agglomerate on Cu. This situation will lead to a sharper Cu-on-Co interface as compared to Co-on-Cu interface. Ag surfactant with $\gamma_{\text{Ag}}=1.24 \text{ J/m}^2$, will help in removing the asymmetry due to its very low surface free energy as compared Cu and Co. In this condition when the growth of the multilayer takes place, the upcoming atoms basically see the lower surface free energy of surfactant atoms instead of other element of the multilayer. This leads to wetting of the upcoming layer. If the surfactant atoms float off to the surface, the deposition of next element will also see the surface free energy of the surfactant. In this situation the layer-by-layer type growth is induced resulting in smooth and symmetric interface in a multilayer.^{18,30}

In our case the obtained results indicate that addition of Ag surfactant is altering the growth mode of the Cu/Co multilayer. The surfactant atoms placed once on the Cu buffer layer balance the surface free energy of Cu and Co resulting in symmetric Cu-on-Co and Co-on-Cu interfaces. As expected, in absence of Ag surfactant, the interface roughness of Co-on-Cu interface is larger as compared to Cu-on-Co interface. An asymmetry in the interface roughness may result in strained interfacial region²⁹ which will act like nucleation centers when the multilayer is annealed at higher temperatures. This will result in enhanced interdiffusion as observed in our case. Whereas since the addition of Ag surfactant removes the asymmetry of the interfaces, such strained regions may be minimized to a large extent resulting in stable interfaces. The reduction of interdiffusion length may be therefore understood in terms of smooth and sharper interfaces formed by addition of Ag surfactant. The XRD results obtained in this work also support this argument as in absence of Ag surfactant a superlattice Cu/Co structure⁴⁶ is formed while no such structure can be observed when Ag surfactant was added.

V. CONCLUSIONS

Therefore in conclusion from the present study we show that addition of Ag surfactant results in smooth and symmetric interfaces in Cu/Co multilayer. The thermal stability of the multilayer improves due to reduced inter-

diffusion.

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